

Diagnostics for the Measurement of Particulate Matter Emissions from Reciprocating Engines

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ABSTRACT

Since 1988, particulate matter emission regulations in the US for heavy-duty diesel engines have mandated a reduction from 0.6 g/bhp-hr to the current level of 0.1 g/bhp-hr. As large an improvement as this has been, however, looming in the not-to-distant future is a requirement for an additional order-of-magnitude reduction, to 0.01 g/bhp-hr by 2007, as illustrated in Fig. 1. It will take a major effort by industry to reach this target, and it will most likely require the use of a particulate trap. But this large reduction in total particulate mass will also create a new problem - how to measure it. The current gravimetric procedure of weighing a sample collected on filter paper will be impractical because of the long time required to collect sufficient mass to be detectable. This problem of measurement sensitivity is compounded by the fact that the size of particles emitted by contemporary engines is far smaller than that of engines of 1988. This is why particulate matter emissions are no longer visible. However, achievement of this reduction in size came at the cost of a tradeoff with the number of particles emitted, which has increased by several orders of magnitude and poses a potential health concern. These new issues of size and number may prove to be as important, or even more so, than particulate mass, raising questions about whether “what” is regulated may also change in the future. Compounding this problem are newly raised issues regarding whether the nanoparticles observed in a dilution tunnel are representative of tailpipe exhaust dilution by the atmosphere. In order for regulators to address these issues, improved measurement techniques are needed now, to provide a better understanding of the importance of size and number on environmental and health issues.

It is also important to note that beginning in 2004, gasoline fueled vehicles will be required meet the same regulations as light-duty diesel vehicles. Current port fuel-injected gasoline engines will have no difficulty meeting the 2004 levels, but it is much less certain for gasoline direct-injection engines, or for either type in 2007. Gasoline engines, in general, are known to emit particulates during cold start, and gasoline direct-injection engines have been shown to produce measurable PM during lean burn operation.

Industry will also need new diagnostic tools to help them meet the 2007 particulate matter requirements. As engine emissions continue to become cleaner due to improvements in the combustion process, the contribution from engine transients will play an ever-increasing role. Only a few of the particulate measurement instruments currently in use respond in real time, and it is doubtful that these will have the sensitivity required for the new regulations.

In this paper, I review the diagnostic tools for particulate matter measurement that are currently available commercially, looking first at those that measure total mass, volume, area, or number, and then those that can characterize particles based on size. I next describe some new techniques that are currently only being used in research, and some that have yet to be demonstrated. I conclude with my recommendations for the instruments most suitable for use today, and my projections for the new techniques that show the most promise.

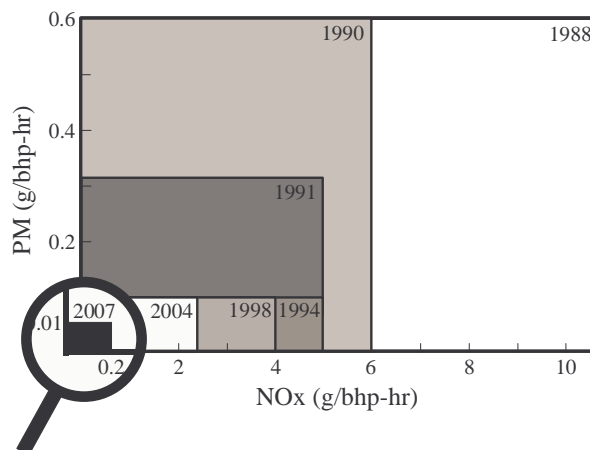


Fig. 1 Evolution of US heavy-duty diesel engine emission regulations.

INTRODUCTION

To introduce this review of measurement techniques for particulate matter (PM) emissions, I first will describe the physical nature of PM and the regulatory measurement procedure prescribed by the US Environmental Protection Agency (EPA) for heavy-duty diesel engines. This will be followed by brief descriptions of instruments that are commercially available. I have separated these into two groups, the first of which I call "global" instruments because the techniques employed provide no information about particle size, and yield only the total mass, volume, area, or number. The second group is reserved for those techniques that classify particles by size. I then close with a description of new optical techniques that show promise, but have not been commercialized, and, in some cases, have not yet been demonstrated.

It is not possible in the limited space available to do justice to the large amount of PM research and application studies that have been reported in the literature. For those seeking a more comprehensive overview, I strongly recommend the series of reports compiled for the US EPA by Prof. David Kittelson and coworkers that can be found at his University of Minnesota web-site [1].

NATURE OF PARTICULATE MATTER

The US EPA has defined PM by virtue of the procedures they have specified for the measurement of engine emissions, as any material deposited on a teflonated fiberglass filter from dilute exhaust gases sampled at a temperature below 51.7 °C. Thus, in addition to carbonaceous material, or soot, PM may also include condensable materials such as sulfuric acid or high boiling point hydrocarbons. (It is not supposed to include measurable amounts of water.) The generally accepted model of engine particulate matter is that it consists of a solid fraction, a soluble organic fraction (SOF), and sulfates. The adjective "soluble" comes from the analytic technique used to extract the organic fraction from the carbonaceous material [2]. The latter two are often combined into a single component, the volatile organic fraction (VOF).

The solid fraction is mostly carbon, which consists of molecules with C/H ratios ranging from 4 to 11. Its density is approximately 2 g/cc. Morphologically, it consists of small, nearly-spherical primary particles 20-50 nm in diameter. These primary particles cluster into chain-like aggregates that can have widely varying sizes, as seen in Fig. 2. Additional solid material occurs in the form of metal ash compounds derived from lubricating oil.

The SOF consists of organic compounds with C/H ratios of 0.5-1.0, with a density of about 0.8 g/cc. This includes the polycyclic aromatic hydrocarbons (PAH), which have 2 to 6 benzene rings joined in various forms. The possible carcinogenic character of PAH is the major cause for the health risk fears of nanoparticle emissions from diesel engines, because these small

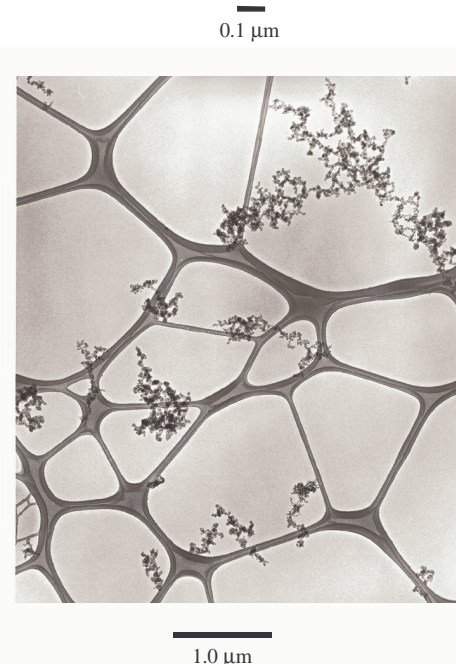
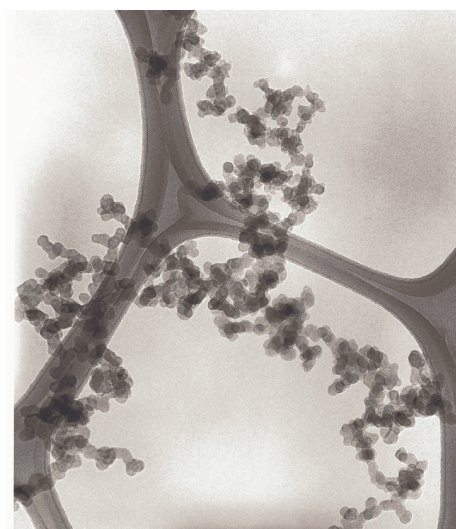


Fig. 2 Transmission electron microscope (TEM) images of soot collected from a diffusion flame.

particles can penetrate deep into lung tissue. Most of the SOF in PM is absorbed onto the surface of the primary particles, where it resides in the form of a thin outer shell. The source of approximately 75% of the SOF is lubricating oil.

The sulfate content of PM is mainly hydrated sulfuric acid that is derived from the fuel sulfur. Sulfur leaves the combustion chamber in the form of sulfur dioxide (>95%) and sulfur trioxide (2-5%) [2]. In the presence of water the latter forms sulfuric acid. The propensity of hydrated sulfuric acid to nucleate is believed to be the main source of the nanoparticles that appear during dilution. Once formed, they act as con-

densation sites for the absorption of unburned hydrocarbons. These small particles are generally free of solid carbon.

REGULATORY PROCEDURE

The US EPA prescribes the exhaust gas sampling and gravimetric (weight change of filter paper for a constant volume sample (CVS)) procedures for diesel exhaust emission measurements in the Code of Federal Regulations [3]. The main difference in the procedures is that double-dilution is a option only for heavy-duty engines. Dilution is necessary to avoid condensation of water, yet permit condensation of SOF on the sample filter. Rather than specify a dilution ratio, the procedure specifies that the exhaust temperature at the sample filter cannot exceed 51.7 °C. It is further specified that the dilution air temperature shall be 25 ± 5 °C.

The specifications on the primary dilution tunnel are that it must be small enough in diameter to cause turbulent flow and of sufficient length to cause complete mixing of the exhaust and dilution air. It also must be at least 46 cm in diameter with single-dilution, or 20 cm with double-dilution.

As long as total mass is the specified parameter for PM regulations, the procedure specified above is adequate. However, if particle size and/or number become regulated in the future, then these specifications will not suffice. Recent studies of particle size distributions in engine exhaust (diesel and gasoline) [4,5,6] have found that residence time and gas temperature can have a large impact on nucleation and the formation of nanoparticles. In order to achieve standardized measurements, it will be necessary to more stringently specify the conditions and dimensions of the dilution system. Even this, however, will not resolve an even larger and more important issue: Do the conditions in the dilution system replicate tailpipe dilution with the atmosphere? The most likely answer to this question is no.

GLOBAL INSTRUMENTS

The tapered element oscillating microbalance (TEOM[®]) [7] is a widely used device for measuring PM mass in real time. A filter substrate is mounted on the tip of a hollow, tapered tube. As the filter collects material, the inertial mass increase alters the natural oscillating frequency of the tube, which is monitored continuously to give a signal proportional to the total mass of the filter. Although the signal can be monitored with high temporal resolution, the sensitivity of the device is limited by the change in mass necessary to create a detectable change in oscillating frequency.

There are a number of techniques that measure PM volume fraction. A smoke number meter [8] measures the loss in reflectivity of a filter as it becomes soiled with PM. Several smoke number scales have been defined, with ranges of both 0-10 and 0-100. A more automated device, capable of continuous operation, is the aethalometer [9]. The optical transmissivity of PM collected on a transparent tape is measured. Periodically, the tape is automatically advanced to a clean section to recycle the process. The opacity meter [10], also called an opacimeter, measures the extinction of light as it traverses the exhaust stream. Because the amount of

extinction is a function of both the PM volume fraction and the path length, the extinction coefficient defined by the Beer-Lambert law is typically used as the parameter to characterize smoke. Laser-induced incandescence (LII) [11] relies on the thermal radiation from laser-heated PM to measure volume fraction and primary particle size. Because non-carbonaceous particles absorb very little energy, while those containing carbon are heated above 4000K, far above the vaporization temperature of any SOF, LII selectively responds to carbon only. Finally, a light-scattering aerosol monitor [12], also called a photometer [13] and nephelometer [14], uses the elastic scattering of light directly from the aerosol to estimate mass concentration. However, because absolute elastic scattering is sensitive to particle size, number, and refractive index, many assumptions are needed to relate the measurement to a physical quantity.

There are three instruments that measure the surface area of particles. The epiphaniometer [15] utilizes a charging chamber to attach lead isotopes to the PM surface. The charged particles are then collected on a filter, from which the measured radioactivity is proportional to the Fuchs surface area. Similar in principle, the diffusion charger (DC) [16] uses a corona discharge to attach positive ions to the PM surface. The particles are collected on a filter, from which the measured current is again proportional to the Fuchs surface area. Lastly, the photoelectric aerosol sensor (PAS) [16] uses ultraviolet light to photoelectrically charge PM by stripping off electrons. Because the propensity for a particle to become charged depends on its composition, this device has been used to selectively measure carbon-bound PAH [17]. By the simultaneous use of a DC and a PAS, it is also possible to distinguish between particles arising from different combustion processes [18].

The condensation nuclei counter (CNC), also called a condensation particle counter (CPC) [19], measures particle number by counting individual particles. Sufficient exhaust dilution is required to reduce the number concentration to $<10^5$ particles/cc to avoid the coincidence of multiple particles in the measurement volume (for higher concentrations, there are correction procedures for multiple particles). The diluted aerosol is saturated with alcohol vapor prior to flowing into a cold condenser, where it is cooled by thermal diffusion. The alcohol condenses onto the particles, growing them into droplets large enough (2-3 μm) to be counted optically as they pass through a laser beam.

The flame ionization detector (FID), commonly used to measure hydrocarbon in engine exhaust, has also been used to measure PM [20]. The presence of carbon particles results in spikes in the FID signal, giving number density. The area under the spikes has been shown to correlate with mass concentration. By using two FID's in parallel, with one filtered to remove all PM, it is possible to make quantitative measurements of hydrocarbon, SOF, and soot.

The global instruments described in this section can also be classified as to whether the measurements are cumulative or instantaneous. This distinction is particularly important with regard to assessing temporal

resolution, because it is the derivative of cumulative measurements that needs to be contrasted with instantaneous measurements. From this perspective, it is only the opacimeter, light scattering, and LII techniques that permit real-time measurements (although LII is limited by the repetition rate of high-energy pulsed lasers). Of these, light scattering is by far the most sensitive technique. (As a point of reference, LII has been estimated to have a sensitivity on the order of a few parts per trillion [21].) The opacimeter is the least sensitive, but there are several ways that this can be improved. Standard practice is to measure the extinction directly across the exhaust flow, such that the path length is limited to the maximum dimension of the tailpipe. However, if the exhaust is sampled through a straight tube with windows at the ends, the path length can be substantially increased [22]. Another approach, which does not require sampling, is to use a multi-pass optical cell to reflect a laser beam multiple times across the exhaust flow [23]. Finally, there is a technique called cavity ringdown [24] where a pulsed laser beam is reflected many times between two mirrors that form a cavity across the exhaust flow. A photodetector behind one of the windows records the very small amount of light that leaks through the mirror ($\sim 0.3\%$) as a function of time for about a microsecond. The decay rate of the signal is a function of the PM concentration.

SIZING INSTRUMENTS

Because of the small sizes of PM emitted by contemporary engines, I will only consider those instruments that can measure particle diameters at least as small as 100 nm. Also, note that the definition of "diameter" depends on the principle of the instrument under consideration.

Cascade impactors are a general class of instrument that use inertial mobility to separate aerosol particles by size. They consist of a stack of discrete stages, each having an orifice or orifices that determine the velocity of gas impinging on an impaction plate. The inertia of large particles causes them to strike and adhere to the impaction plate, while smaller particles flow around the plate to the next stage; each successive stage captures particles of a smaller size range. A micro-orifice uniform deposit impactor (MOUDI) [25] uses multiple orifices and rotating impactor plates to optimize the uniformity of the collected sample, to facilitate subsequent analysis of its composition or morphology. The impaction plates are easily removed for the installation and removal of foil or filter substrates for the collection of the PM samples. There are typically about ten stages with a size range from 56 nm to 10 μm . Smaller sizes down to 10 nm can be classified with a nano-MOUDI, which uses low pressure on the last few stages.

An electrical low pressure impactor (ELPI) [26] is an inertial particle sizer with an automated readout capability. A corona discharge is used to charge the particles with positive ions. The impactor plates that they collect on are electrically isolated, permitting the current from each to be read with an electrometer. A typical

device will have twelve stages for sizes ranging from 30 nm to 10 μm .

The electrical aerosol analyzer (EAA) [27] uses electrical mobility to classify particle size, and at one time it was the most commonly used instrument for diesel PM measurements. As a result, there are numerous references to its use, both past and present, even though it is obsolete and has been commercially unavailable for more than ten years.

The differential mobility analyzer (DMA) [28] is now considered the device of choice for classifying particles by electrical mobility. It consists of two concentric metal cylinders. The inner cylinder, the collector rod, is maintained at a controlled negative voltage, while the outer cylinder is electrically grounded, creating an electric field that attracts the particles to the collector rod. Particles in the polydisperse aerosol flowing along the annulus precipitate along the rod, the location depending on the particle electrical mobility. At a small slit at the end of the rod, particles with a narrow range of electrical mobility exit the DMA as a monodisperse aerosol flow.

A differential mobility particle sizer (DMPS) [29] is simply a DMA used together with the previously described CPC to measure the number concentration in the monodisperse aerosol flow. By varying the voltage on the collector rod, a particle size distribution can be created. For a scanning mobility particle sizer (SMPS) [30], the collector rod voltage is dynamically scanned to produce a complete size distribution in as little as one minute.

For real-time measurements of particle size distribution, the ELPI is the only instrument available. However, because the number of size ranges is limited with the ELPI, the SMPS is the instrument of choice when real-time is not a requirement. It is also possible to run the SMPS in a fixed-size mode in real-time to create a size distribution from an ensemble of repeated tests. If composition or morphology of the PM is desired, then the MOUDI is the instrument of choice.

RECENT OPTICAL TECHNIQUES

All of the techniques presented thus far are available as commercial instruments. There are, however, a number of optical techniques still in the development stage that show promise as new tools for PM studies.

The scatterometer [31], an angle-scanning polarization-modulated nephelometer, completely characterizes laser light scattered from an aerosol using Mie theory. Information concerning the size distribution, complex refractive index, and particle shape can be obtained. The technique has been successfully applied to diesel exhaust by Hunt et al. [32].

The LII technique discussed earlier, when combined with laser elastic scattering (LES) measurements interpreted with Rayleigh-Debye-Gans polydisperse fractal aggregate (RDG/PFA) theory [33], can provide useful estimates of aggregate parameters. The quantities that can be determined include particle volume fraction, primary-particle size and number density, aggregate surface area and size distribution (concentrations of aggregates having a specific number of primary parti-

cles), and the fractal dimension, a measure of particle-shape irregularity. The procedure has been successfully applied to the study of soot in a stationary flame by Köylü [33].

Laser-induced vaporization (LIV) appears to be a potentially useful tool for the real-time, in situ investigation of carbon-bound SOF. Case and Hofeldt [34] suggest that the energy consumed by LIV of SOF will cause a time delay in LII signals. If the LII procedure of Snelling et al. [35] is used, the particle temperature will also be known, permitting a correlation with temperature that could provide insight into the SOF composition. Witze et al. [36] have shown that time-resolved LES measurements can be used to estimate particle size change from LIV of soot (LIVES). The procedure described would appear to be applicable to the measurement of carbon-bound SOF. In addition, because non-carbonaceous PM will scatter but not absorb laser light, simultaneous LII and LES has the potential to distinguish between non-carbonaceous and carbonaceous PM. Finally, while there is a convenience benefit for using a single, pulsed laser for LIV studies, there also could be a significant advantage for using a second, cw laser for the LES. Time-averaged LES measurements for a microsecond just before and after the laser pulse would be sufficiently fast to freeze the measurement volume and yet avoid background problems from the pulsed laser, LII, and laser-induced fluorescence (LIF). [37] have demonstrated excimer laser fragmentation-fluorescence spectroscopy (ELFFS) to investigate PM in diesel exhaust. High-energy laser pulses are used to photofragment soot to form atomic carbon. Once formed, the carbon is then induced to fluoresce with remaining energy from the same laser pulse; the fluorescent signal is proportional to the mass concentration of carbon. This technique has the potential for measuring any species that will fluoresce.

Laser-induced breakdown spectroscopy (LIBS) is a well-established technique used to measure the composition of gases, liquids, and solids. It has recently been demonstrated by Buckley et al. [38] as a continuous monitor for toxic metal emissions from incinerators. A focused, high-energy laser beam is used to create a spark (breakdown) in the medium being investigated. In gases, the extremely high temperatures in the resulting plasma lead to dissociation of molecules into their constituent atoms, and excitation into excited electronic states. As the plasma cools, relaxation back to the ground state is accompanied by light emission at species-specific wavelengths that is quantifiable.

SUMMARY RECOMMENDATIONS

Because current PM regulations identify mass as the parameter of concern, and specify that it be measured by the gravimetric technique, this has to be considered the preferred procedure for steady-state measurements for the present time. For transient tests where several seconds of response time is sufficient, the TEOM is suitable for today's regulations. However, it lacks adequate sensitivity for the low PM levels mandated for the future.

For fast transients, the instantaneous volume measurement capability of the opacimeter is ideal for the present time. However, it also lacks the necessary sensitivity for the future regulations. Too little is known about the improvements that can be achieved by either multi-pass optics or cavity ringdown techniques to know how well these compare with LII, but the restrictive response of the latter to only carbon may be considered a liability.

If at some time in the future particle number becomes regulated, the CPC is capable of fulfilling the need. If particle size becomes regulated, the ELPI is an efficient real-time instrument for the larger particles, but cannot resolve nanoparticles. The latter can be measured with both the nano-MOUDI and the SMPS, but because the measurement of the former is gravimetric, while the latter uses counting, the SMPS is superior for very low PM levels.

Of the new optical techniques described, I feel that fluorescence and Mie scattering theory may be too complex for this application, and believe that the combined use of LII, LIV, and LIBS with LES offers the best opportunity for success. These are compatible techniques with the potential to characterize the size, shape, and composition of the irregularly-shaped particles in engine exhaust.

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